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, NEW YORK, USA

The following men attended the conference:

Lt. Col. A. E. Buffard	Technical Services Division
Capt. J. P. Finley	"
Capt. G. W. Russell	"
Mr. J. S. Livingston	Consolidated Chemical Company
Dr. R. H. Cramphor	"
Dr. R. M. Bitchens	"
Mr. W. D. Brewster	"
Mr. J. P. Stickley	"
Mr. W. J. Wigand	"

INTRODUCTION

Lt. Colonel Buffard, visiting this plant from Washington, explained his position and the purpose of his visit. Colonel Cramphor is Chief of the Technical Services in the Office of the Chief, Chemical Warfare Service. Lt. Colonel Buffard is Chief of Research and Development. Col. Buffard made the following points as background for the conference.

1. Due to experiences at the Niagara Falls plant, 1943, it has been decided that CCP manufacture is more of a research and development problem than a production problem. The problem of continuation of research and development in the four plants has been given to Colonel Buffard. It is his responsibility to see when the plants are operating properly and then release technical control to the manufacturing division.

2. Even though the plants are in the research and development stage, it is planned to operate the Newark, Holland and N. Y. plants at an early productive capacity as possible and use the Niagara Falls plant as a test and scale pilot plant.

3. Colonel Buffard had his civil service experience from the Drewrywood Arsenal visit Niagara Falls plant to report to the Chemical Warfare and hold a conference with the plant officials in Niagara to determine the status of the present "Newark" I & II sections. Colonel Buffard also held a conference at Newark plant to meet their officials and opinions on CCP production. At the same time he discussed the Lewis Hunt and Holland plant to return to my information which he had obtained and to discover from the working and processes used at these two plants, any items which may have been omitted.

4. Colonel Buffard very sincerely explained that in the solution of the general CCP problem he and only at the present time.

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1. In addition to the 100 ton/hour, 30% conversion plant, the production of CO<sub>2</sub> will be increased by 200 ton/hour. This will be done by adding two additional 100 ton/hour units to the existing plant. The new plant will be located at the same site.

2. The 100 ton/hour plant of the first half of January 1968 has been an evaluation study of the conversion process with test samples in an laboratory and pilot plant scale. The final evaluation is a list of recommendations, some of which seem either impractical or require too high a cost in dollars or in physical details.

3. Static extraction problems will be considered under the various stages of the process.

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4. Colonel Haafford asked Monsanto Chemical Company to consider the possibility of using the DIT process in our present plants. The reason for using the DCA process is that the CO<sub>2</sub> produced has twice the life of DCA-CO<sub>2</sub> in storage conditions. Extreme stability of the finished product is no longer required since the material is immediately needed for immediate use.

5. Edgewood Arsenal has converted their CO<sub>2</sub> plant to the production of the second and third steps of the DIT process. They take CO<sub>2</sub> in another plant and use 12 of the 16 reactors for the second step and 4 reactors for the third step. The bottleneck in this type of plant is the refrigeration capacity for cooling Step-II. Edgewood Arsenal is installing additional refrigeration capacity.

6. Edgewood Arsenal has approached the possibility of building a 100 ton/hour plant and adding it to the other carbon dioxide production facilities to take care of all. This is not a straightforward or simple solution. However, at the present time, the four plants in use are not able to handle the present load. However, the problem can be solved by adding a plant in another place and returning to the plant the CO<sub>2</sub> from the plant. It is also possible to convert the existing plant to the DCA process as well. Colonel Haafford is requesting with regards to the DCA process for early conversion. Although it is not unusual to convert a plant to a different process, the present bottleneck is the lack of time and a cost factor. It is believed to be feasible to accomplish this in a reasonable time.

7. There are several ways to accomplish the conversion of the existing plant.

8. The first method would be to add the necessary equipment to the existing plant to convert it to the DCA process. This would be a costly conversion. The second method would be to add a new plant to the existing plant. The costs of the new plant would be dependent upon the size of the new plant.

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only a few months. Also, it is very difficult that we would be able to obtain the critical metals to replace the solvent recovery equipment even if it were feasible from the standpoint of costs.

b. After several months operation, Niagara Falls Plant is using 4 pounds of 3d per pound of CO<sub>2</sub> produced while at Lawrence Arsenal where they are running on the DPF process they are only using 1.1 pounds of 3d per pound of CO<sub>2</sub> produced. According to the information Colonel Bradford has obtained from NPA it may be impossible to obtain enough 3d to keep the TCA plants running.

NOTES ON CORROSION

1. Twelve batches per day are required to obtain the rated plant capacity of 20,000 pounds per day of finished product. This can be made in the present equipment.

2. It takes a long time to wash the 3d to a low acid limit in the filter press. At present Niemants is running an eight hour cycle in the process which gives him four hours working time. If the low acid limit has not been obtained, Niemants has been washing the 3d in the melt tank by adding water, ammonia and ammonium. Niemants has also installed a valve of venting the filter cake from the upper melt head port. This is connected to the filter cake drain line. Under these conditions the pressure at the head of the press is approximately 5 pounds until the press is almost full when the pressure rises to 15 pounds.

3. According to Niagara Falls Plant the melt tanks are the only place of corrosion in the process equipment. It is believed that Niemants's practice of completing the washing of the 3d to a low acid limit in the melt tanks may reduce this corrosion. Colonel Bradford requested Niemants to inspect the melt tank they have been using and teletype a report to him at Midland on Monday, March 12th.

Notes:

The melt tanks were inspected on Monday as requested. 32 batches had been processed through the tank before the inspection was made. It was found that the spot bonded lead lining had buckled and pulled away from the sides of the steel tank in two places. Metal corrosion was very slight but was apparent.

Niagara Falls Plant is flanging the melt tanks and using sheet lead to replace their homogeneous lead lining. They have also replaced the lead lining with an experimental acid proof brick lining. No results are available on this latter experiment.

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4. It was felt that the formation of the so called red oil is the formation of the so called red oil. The instability is difficult to remove and is undoubtedly carried through to the finished product and may be one of the causes of the poor stability of the Niagara Falls product compared to the pilot plant CO<sub>2</sub> made by the N.Y. process. The red oil has also been definitely established as the main source of corrosion in the Step-I evaporators. Three possible ways of reducing the formation of the red oil were suggested.

a. Use caustic hydrochloride as a raw material instead of sodium.

b. Edgewood Arsenal suggests running Step-I at a lower temperature, that is, at a maximum of 20° C. This suggestion brings up the fact that the refrigeration system is either inadequate to maintain the correct temperature on all reactors at the same time, or the cooling water is not going into the reactor jacket fast enough. If the latter is the case, it might be corrected by placing a booster pump in the inlet cooling water line or by increasing the rate of agitation in the reactors from 100 to 150 rpm to prevent local overheating.

c. Increase the amount of red wash, thereby increasing the amount of Step-II distillation. In order to do this it would be necessary to strike a balance between the amount of red redistilled and the solubility of trichloroethylene hydrochloride in S.C.

5. Monsanto believes the vent lines on the reactors are unsatisfactory because they are too small. They expect to increase the size and also may have to make a change in the absorption system. They do not believe that the vent cause absorption system will be adequate when the production rate is increased. In this connection Edgewood Arsenal is building large vertical packed caustic absorption towers.

6. Monsanto is working on an experimental plan to distill either Ni or Cu from all stages of the process and the weak filtrate in one of the existing glass lime reactors and then redistill it in the copper evaporator. Niagara Falls suggests that Fastaloy C is no better than copper in the presence of Step-I filtrates but silver and tantalum are satisfactory.

7. It was suggested that a solenoid line or valve be used between the Step-I reactor valve and the slurry pipe.

8. Orders were received at this plant not to attempt to operate the incinerator until a satisfactory procedure had been determined at the Niagara Falls plant. Since no further work has been done on this problem at Niagara Falls, Colonel Hafford requested Monsanto to attempt to evolve a satisfactory method of sludge disposal in the incinerator. It was commented that the sludge from the evaporator while still in a liquid form be dissolved in a low grade fuel oil and atomized through an oil burner. It may be necessary to heat the

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surfaces at incandescence by supplying additional natural gas. This procedure would depend on the solubility in the alkyne in the final oil. It is also understood that when the Laskerator is used it will probably be necessary to replace the steel chimney with brick.

2. The control system for feeding hot water to the filter presses does not work. It is planned to install a surge tank and control the temperature of the water in the tank.

NOTES ON STEP-II

1. Monocro has made a few minor changes in Step-II equipment. They have placed an air coupling on the Rd manifold from the melt tanks to the reactors. They have established a practice of bleeding the Rd line with air after each batch of Rd is charged to the reactor. Monocro has also reversed the water flow in the 100 square foot condenser. It now enters at the bottom and discharges from the top of both the exterior and interior jackets. The Rd distilled from the Step-II reactor during the reaction is now trapped out of the vent line and returned directly to the filtrate tank.

2. It is well known the Step-II centrifuges are the bottleneck for plant production. The slurry centrifuges are faster than the rubbery centrifuges but under best conditions it would be impossible to obtain more than 50,000 pounds per day from the present equipment. It is possible to load and wash the centrifuges while running the tanks at top speed but the washing to remove the monochloroaldehyde is very slow. It requires a 1½ hour water wash and a ½ hour drying period. In view of this situation Mr. Stickley would like to know the basis for comparison and for figuring the additional filter press capacity which is being installed at Niagara Falls Plant. Colonel Bufford will endeavor to obtain these figures from Biltmore and forward them to Mr. Stickley.

3. Edgewood Arsenal believes that one of the reasons for the lower stability of the TGA process product is that too much chloroacetalide is left in the product during the Step-II filtration. Chloroacetalide is soluble in DCE and Rd. If the assumption is correct, the chloroacetalide could be removed either with a hot Rd wash or by dropping the charge from the reactor at a higher temperature. Present practice is to cool the Step-II reactor charge to 10° C and then circulate the charge up to the centrifuge and back to the reactor until it has all been charged to the centrifuge. This substantially lowers the temperature of filtration far below the 10° C temperature at which it is discharged from the reactor. Dropping the reactor charge at a higher temperature will be tried at this plant but it is believed it will create a circulation problem around the centrifuge.

4. The Midland Plant partially confirms the above argument in a recently reported where they state that a thin insulating layer was found next to the centrifuge basket through which it is virtually impossible to wash. Mr. Garath also confirms that the product in the laboratory is much easier to filter than the plant product, indicating that there are some byproducts in the plant product which reduces the washing permeability of Rd.

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5. Moncato reports that UNCLASSIFIED the Step-II solvent. Carbon carrying solvent to the Step-II reactor may have been determining the amount of  $\text{Pd}$  in the solvent and reducing the  $\text{Pd}$  charge by that amount.

6. Dr. Garrath reports that 50% of the urea charged to the Step-II reactor is converted to  $\text{CO}_2$ . The conversion is 50% regardless of the amount of urea charged. This large gas discharge through the 100 square foot condenser is undoubtedly the reason for the  $\text{CO}_2$  being carried over and out the vent system or returned to the filtrate tank.

7. The  $\text{Pd}$  is dried at  $130^\circ \text{C}$ . Since the sublimation temperature of  $\text{Pd}$  is  $75^\circ \text{C}$  some of the  $\text{Pd}$  impurity in the  $\text{Pd}$ -2 undoubtedly leaves during the drying period.

8. The drawings for the new Step-II facilities show a caustic tank. It is not known whether the caustic tank is for use in Step-II or for washing the Step-I filter press.

NOTES ON STEP-III

1. At the suggestion of Edgewood Arsenal production men, a telltale has been installed on the Step-III reactor slurry manifold. Colonel Rutherford stated that  $\text{NaCl}$  is the principle impurity in  $\text{CO}_2$  and it is believed that any purification treatment subsequent to the completion of the final product reduces the stability of the final product. From these facts two important questions are apparent:

a. In the Step-III chlorination of the  $\text{NaCl}$  is there an equilibrium established which would cause the  $\text{CO}_2$  loss chlorine after the chlorination has passed a certain point?

b. Since the  $\text{NaCl}$  is not soluble in  $\text{CO}_2$  the Step-III reaction is an attempt to chlorinate a slurry. In order to obtain intimate contact for the chlorination, it is imperative to have a small particle size in the slurry. At present nothing is known concerning the particle size of the  $\text{NaCl}$  as charged to the Step-III reactor. Both of the above two points will be investigated by the Moncato research men.

EVALUATION

11. Dr. Garrath has obtained the following figures on the corrosion rate of carbon in the bottoms of the various  $\text{Pd}$  filtrates used in the plant.

Solvent	Inch Penetration per Month
Beak filtrate	.38
Step-I	.09
Step-II	.04
Step-III	.01

According to Niagara Falls qualitative data Step-II corrosion is as bad as



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Step-I and Step-II corrosion is just as bad as Step-I and the latter can be treated effectively prior to distillate. Niagara Falls now believes that the weak filtrate corrosion in the azeotropic still is not as bad as any of the strong liquors. They plan on starting to use the azeotropic still again as soon as a new set of heating tubes is obtained.

2. The Office of the Chief, CGS, has ordered an extra copper tube bundle for each of the four plants. Since Metaloy C is satisfactory for Step-II a Metaloy C tube bundle has also been ordered for each plant.

3. It is believed that the zinc treatment of the Step-I filtrate does not give a satisfactory result when the zinc is used in reasonable quantities. When the operating directive quantities are used Dr. Gorenth found that Step-I filtrate treated with zinc produces a corrosion rate of .05 inches penetration per month on copper. It is, therefore, assumed that zinc is not satisfactory for Step-I.

4. Due to lack of information on a better method this plant is at present using sodium bisulfite as the treatment for Step-II filtrate and no treatment for the Step-I filtrate.

~~ENCLOSURE~~

1. Mr. Stichley estimates that a production rate of 60,000 pounds per month can be obtained immediately and believes it will be possible to produce 70,000 pounds per month with only a few minor improvements.

G. W. HENKEL,  
Captain, CGW.R.C.,  
Executive Officer

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ADDENDUM TO

REPORT OF CONFERENCE AC ST. LOUIS PLANT

CHEMICAL WARFARE SERVICE

Friday, March 20, 1942

March 23, 1942

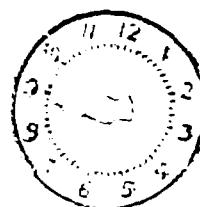
1. Reference paragraph 6, page 4; it was incorrectly stated in the original report that Monsanto Chemical Company plans to re-distill the mother liquor and weak filtrate in copper evaporators after the initial distillation in the glass line reactor. A single distillation from the glass line reactor is planned, except in the case of the weak filtrate which will be concentrated in the azeotropic still.

2. Reference paragraph 2, page 5; substitute ammonium sulfate for the words ammonium bisulfite.

3. Reference paragraph 5, page 6; since the conference, Monsanto Chemical Company has found that reducing the P-1 charge to the Step-II reactor by the amount of P-1 built up in the re-circulated solvent, reduces the P-2 batch yield. Therefore, this practice has been discontinued and a normal amount of P-1 is charged to the Step-II reactor.

G.W.C.W.

J. W. MCGILL,  
Capt., C.S.C.M.C.P.  
Executive Officer 3/20/1942



Chemical Warfare Service